Monatsh Chem 139, 1457–1461 (2008)
DOI 10.1007/s00706-008-0954-9
Printed in The Netherlands

Monatshefte für Chemie
Chemical Monthly

Regioselective hydrolysis of ketenimines derived from NH-acids and acetylenic esters in the presence of *tert*-butyl isocyanide under neutral conditions

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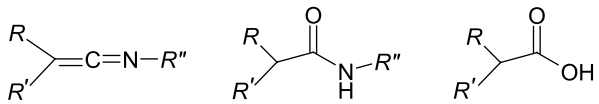
Received 24 March 2008; Accepted 1 May 2008; Published online 26 June 2008

Abstract The regioselective hydrolysis of ketenimines derived from NH-acids, such as 2,2,2-trichloro-*N*-phenylacetamide or ethyl-2-anilino-2-oxoacetates and acetylenic esters in the presence of *tert*-butyl isocyanide in a THF/H_2O system (1/1) without any catalysis leads to a diastereomeric mixture of dialkyl 2-[(*tert*-butylamino)carbonyl]-3-[(2,2,2-trichloroacetyl)anilino]succinates and dialkyl 2-[(*tert*-butylamino)carbonyl]-3-[2-ethoxy-2-oxoacetyl)anilino]-succinates in good yields. Dynamic NMR effects were observed in the ^{13}C NMR spectra of diethyl 2-[(*tert*-butylamino)carbonyl]-3-[(2,2,2-trichloroacetyl)anilino]succinate as a result of restricted rotation around the *N*-aryl single bond. The free energy of activation ($\Delta G^{\#}$) for this process is 37.9 kJ mol^{-1} which leads to an observable atropisomerism.

Keywords Hydration; Heteroallenes; Hindered rotation; Atropisomers.

Introduction

In recent years, the synthesis applications of multifunctional heteroallenes have been widely investigated. These heterocumulenes were particularly useful in the synthesis of nitrogen-containing heterocycles through their participation in electrocyclic ring closures [1], intramolecular [2 + 2] and [4 + 2] cycloadditions [2, 3], and imino-ene type reactions [4]. There are many theoretical studies on the hydration of ketenimines but little attention has been paid to the hydrolysis of new synthetic multifunctional ketenimines [5–7]. On the basis of *Hegarty et al.* [8] studies, the hydration of ketenimines **1** to give hydrolysis products **2** and **3**, shows dependence on hindrence of substituted groups on ketenimines and *pH* of the reaction conditions.



1 **2** **3**

Scheme 1

We have previously reported the synthesis of highly functionalized ketenimines from the reaction of some NH-acids and acetylenic esters in the presence of alkyl isocyanides [9–13]. We now focused our attention on the hydrolysis of ketenimines derived from NH-acids, such as 2,2,2-trichloro-*N*-phenylacetamide or ethyl 2-anilino-2-oxoacetates and acetylenic esters in the presence of *tert*-butyl isocyasnide in THF/H_2O system in neutral condition to produce new multifunctional compounds **5a**–**5f** in good yields (see Scheme 2).

Results and discussion

Three-component reaction of *tert*-butyl isocyanide with electron deficient acetylenic esters in the pres-

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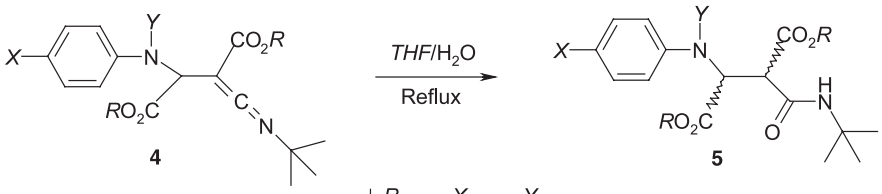


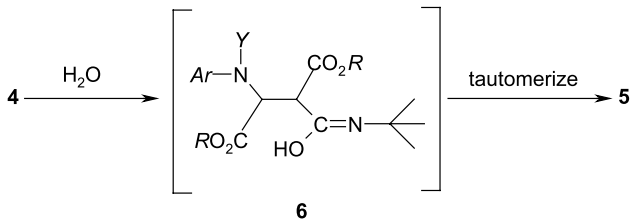
	R	X	Y
4a, 5a	*Me*	H	CCl_3
4b, 5b	*Et*	H	CCl_3
4c, 5c	*Me*	H	$COCO_2Et$
4d, 5d	*Me*	Br	$COCO_2Et$
4e, 5e	*Me*	NO_2	$COCO_2Et$
4f, 5f	*Et*	NO_2	$COCO_2Et$

Scheme 2

ence of strong NH-acids at ambient temperature in CH_2Cl_2 leads to ketenimines **4** [9, 10]. Subsequent hydrolysis of these ketenimines in the THF/H_2O (1/1) system without any catalysis leads to a diastereomeric mixture of multifunctional compounds **5a**–**5f** in good yields (Scheme 2).

The structures of **5a**–**5f** were deduced from their elemental analysis and IR, ^{1}H NMR, and ^{13}C NMR spectroscopic data. The mass spectra of these compounds displayed molecular ion peaks at appropriate m/z values. Due to the presence of two chirality centers in **5**, the NMR spectra of these compounds are consistent with the presence of two diastereomers. For example the ^{1}H NMR spectrum of **5a** is consistent with the presence of two diastereomers in 66:34 ratios. The ^{1}H NMR spectrum of the diasteromeric mixture of **5a** exhibited six sharp lines for *tert*-butyl ($\delta = 1.39$ and 1.46 ppm) and methoxy ($\delta = 3.81$, 3.84, 3.85, and 3.86 ppm) protons. The NH proton appeared as two broad lines at ($\delta = 5.67$, and 5.83 ppm) and the aryl moiety appeared as multiplet at $\delta = 7.31$–7.85 ppm. Two aliphatic CH protons of **5a** exhibited four dublets ($\delta = 4.31$, 4.38, 5.03, and 5.20 ppm, $^3J_{HH} = 10.1$ Hz). The ^{13}C NMR spectrum of the diasteromeric mixture of **5a** exhibited 34 resonances in agreement with the proposed structures. In case of **5b**, the major isomer can be separated by column chromatography. We were not able to isolate the minor isomer of **5b** in pure form; however, its NMR data can be extract from the mixture of the two diastereomers. Partial assignments of these resonances are given in Experimental Section.

A proposed mechanism for this reaction is outlined in Scheme 3 based on the previous reports



Scheme 3

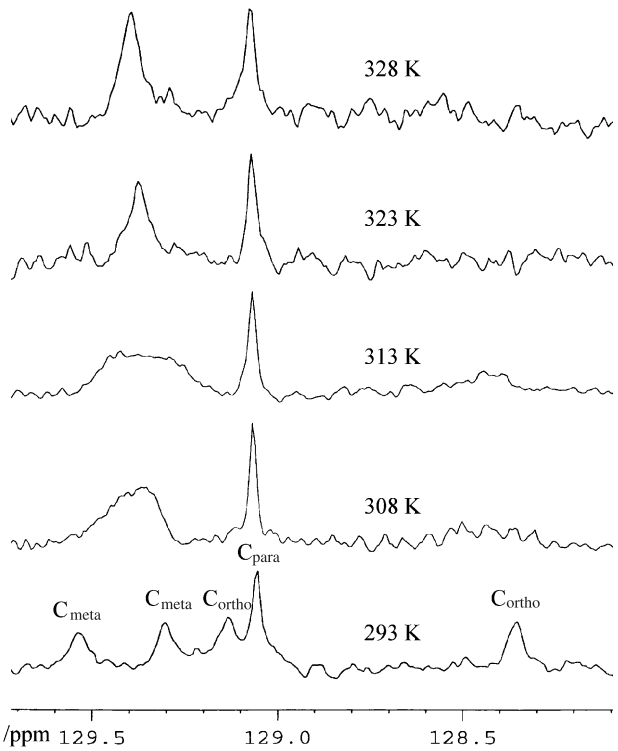
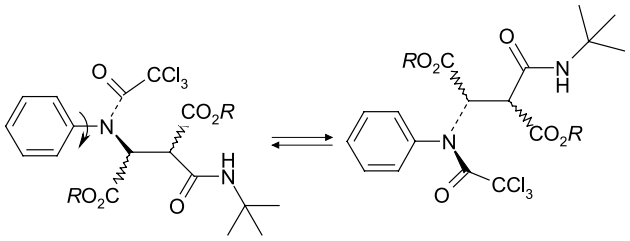


Fig. 1 Variable temperature 62.9 MHz ^{13}C NMR spectra of **5b** in $CDCl_3$



Scheme 4

[5–8]. Initial addition of water to ketenimines **4** leads to intermediates **6**. Such an addition product may tautomerise to amide **5** (Scheme 3).

The benzene region of the ^{13}C NMR spectra of **5a** and **5b** in $CDCl_3$ at room temperature exhibits four broad signals for the ortho and meta aromatic CH groups (see Fig. 1). Increasing the temperature to about 40°C results in coalescence of CH_{meta} resonances. At 55°C the meta carbon atoms appear as a relatively sharp single resonance. This dynamic NMR effect is attributed to slow rotation around the phenyl–N bond in **5a** and **5b** (Scheme 4). Hindered rotation around the N–CO bond in **5a** and **5b** is ruled out, because only one signal is observed for the para (or ipso) carbon atom of the phenyl ring at various temperatures.

Although an extensive line-shape analysis in relation to the dynamic ^{13}C NMR effect observed for **5a** and **5b** was not undertaken, the variable temperature spectra of **5b** allowed calculating the *Gibbs* energy barrier (if not the enthalpy and entropy of activation) for the dynamic NMR process. From coalescence of the meta carbon resonances and using the expression $k = \pi\Delta\nu/\sqrt{2}$, we calculated that the first-order rate constant (k) for dynamic NMR effect in **5b** is $27\ s^{-1}$ at 40°C. Application of the absolute rate theory with a transmission coefficient of 1 gives a *Gibbs* energy of activation ($\Delta G^{\#}$) of $37.9 \pm 2\ kJ\ mol^{-1}$, where all known sources of errors are estimated and included [14]. The experimental data available are not suitable for obtaining meaningful values of $\Delta H^{\#}$ and $\Delta S^{\#}$, even though the errors in $\Delta G^{\#}$ are not large [15].

In conclusion, the regioselective hydrolysis of ketenimines derived from NH-acids such as 2,2,2-trichloro-*N*-phenylacetamide or ethyl 2-anilino-2-oxoacetates and acetylenic esters in the presence of *tert*-butyl isocyanide in a *THF*/H_2O system leads to a diastereomeric mixture of dialkyl 2-[(*tert*-butylamino)carbonyl]-3-[(2,2,2-trichloroacetyl)anilino]succinates and dialkyl 2-[(*tert*-butylamino)carbonyl]-3-[2-ethoxy-2-oxoacetyl)anilino]succinates in good yields. Dynamic NMR effects are observed in the ^{13}C NMR spectra of **5a** and **5b** and are attributed to restricted rotation around the aryl-nitrogen bond.

Experimental

Ketenimines were prepared by known methods [9, 10]. Melting points were measured with an Electrothermal 9100 apparatus. Elemental analyses of C, H, and N were performed using a *Heraeus* CHN-O-Rapid analyzer. These results agreed favorably with the calculated values. IR spectra were measured with a Shimadzu IR-460 spectrometer. NMR spectra were recorded with a Bruker DRX-250 AVANCE instrument (250.1 MHz for ^1H and 62.9 MHz for ^{13}C) with $CDCl_3$ as solvent. Chemical shifts are given in ppm (δ) relative to internal *TMS*, and coupling constant (J) are reported in Hertz (Hz). Mass spectra were recorded with a Finnigan-Matt 8430 mass spectrometer operating at an ionization potential of 70 eV.

*General procedure (examplified by **5a**)*

To a magnetically stirred solution of 0.48 g 2,2,2-trichloro-*N*-phenylacetamide (2 mmol) and 0.28 g dimethyl acetylenedicarboxylate (2 mmol) in 6 cm^3 CH_2Cl_2 were added drop-wise at −10°C over 10 min 0.45 g *tert*-butyl isocyanide (2 mmol) in 2 cm^3 CH_2Cl_2. The reaction mixture was then allowed to warm up to room temperature and stirred for 24 h. The solvent was removed under reduced pressure and 10 cm^3 *THF*/H_2O (50/50) were added to the reaction mixture and it was then refluxed for 18 h. The solvent was removed under reduced pressure, and the residue was purified by silica gel (Merck 230–400 mesh) column chromatography using *n*-hexane/*Et*OA*c* mixture as eluent.

Dimethyl 2-[(tert-butylamino)carbonyl]-3-[(2,2,2-trichloroacetyl)anilino]succinate (**5a**, $C_{19}H_{23}Cl_3N_2O_6$)

Yellow powder, mp 138–140°C; yield 0.52 g (54%); IR (KBr): $\bar{\nu} = 3495$ (NH), 1753, 1678 (C=O) cm^{-1}.

Major isomer (66%); ^1H NMR (250.1 MHz, $CDCl_3$): $\delta =$ 1.46 (s, C*Me*$_3$), 3.81, 3.85 (2s, 2OCH_3), 4.38, 5.03 (2d, $^3J_{HH} = 10.1$ Hz, 2CH), 5.83 (s, br, NH), 7.31–7.85 (m, H–*Ar*) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$): $\delta = 28.6$ (C*Me*$_3$), 52.3, 52.9 (2OCH_3), 53.1, 54.7 (2CH), 67.4 (*C*Me$_3$), 92.0 (CCl_3), 128.6, 128.8 (2C_{ortho}), 129.1 (C_{para}), 129.2, 129.4 (2C_{meta}), 142.6 (C_{ipso}), 162.3, 164.4, 168.2, 168.9 (4C=O) ppm.

Minor isomer (34%); ^1H NMR (250.1 MHz, $CDCl_3$): $\delta =$ 1.39 (s, C*Me*$_3$), 3.84, 3.86 (2s, 2OCH_3), 4.31, 5.20 (2d, $^3J_{HH} = 10.1$ Hz, 2CH), 5.67 (s, br, NH), 7.31–7.85 (m, H-*Ar*) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$): $\delta = 28.5$ (C*Me*$_3$), 51.9, 52.8 (2OCH_3), 53.6, 54.2 (2CH), 66.7 (*C*Me$_3$), 92.1 (CCl_3), 128.3, 128.5 (2C_{ortho}), 129.0 (C_{para}), 129.3, 129.5 (2C_{meta}), 141.9 (C_{ipso}), 162.2, 164.1, 168.0, 168.8 (4C=O) ppm.

Diethyl 2-[(tert-butylamino)carbonyl]-3-[(2,2,2-trichloroacetyl)anilino]succinate (**5b**, $C_{21}H_{27}Cl_3N_2O_6$)
Yellow oil, yield 0.53 g (52%).

Major isomer (75%), white powder, mp 126–127°C; IR (KBr): $\bar{\nu} = 3460$ (NH), 1747, 1682 (C=O) cm^{-1}. ^1H NMR (250.1 MHz, $CDCl_3$): $\delta = 1.28$, 1.34 (2t, $^3J_{HH} = 7.3$ Hz, $2OCH_2CH_3$), 1.43 (s, CMe_3), 4.20–4.37 (m, $2OCH_2CH_3$), 4.36, 4.97 (2d, $^3J_{HH} = 10.3$ Hz, 2CH), 5.82 (s, br, NH), 7.33–7.85 (m, H–*Ar*) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$): $\delta = 14.0$ ($2OCH_2CH_3$), 28.6 (CMe_3), 52.2, 54.8 (2CH), 61.7, 62.3 ($2OCH_2CH_3$), 67.4 (CMe_3), 92.0 (CCl_3), 128.4, 129.1 ($2C_{ortho}$), 129.0 (C_{para}), 129.3, 129.5 ($2C_{meta}$), 142.7 (C_{ipso}), 162.2, 164.7, 167.7, 168.4 (4C=O) ppm.

Minor isomer (25%); ^1H NMR (250.1 MHz, $CDCl_3$): $\delta = 1.22$–1.42 (m, $2OCH_2CH_3$, CMe_3), 4.20–4.37 (m, $2OCH_2CH_3$, CH), 5.11 (d, $^3J_{HH} = 10.0$ Hz, CH), 5.64 (s, br, NH), 7.19–7.85 (m, H–*Ar*) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$): $\delta = 13.8$ ($2OCH_2CH_3$), 30.3 (CMe_3), 51.9, 54.7 (2CH), 61.3, 63.0 ($2OCH_2CH_3$), 67.3 (CMe_3), 92.8 (CCl_3), 128.3, 129.8 ($2C_{ortho}$), 129.2 (C_{para}), 129.7, 129.9 ($2C_{meta}$), 142.7 (C_{ipso}), 162.1, 164.6, 167.5, 168.3 (4C=O) ppm.

Dimethyl 2-[(tert-butylamino)carbonyl]-3-[(2-ethoxy-2-oxoacetyl)anilino]succinate (**5c**, $C_{21}H_{28}N_2O_8$)
Brown powder, mp 136–137°C, yield 0.45 g (51%); IR (KBr): $\bar{\nu} = 3380$ (NH), 1742, 1670 (C=O) cm^{-1}.

Major isomer (89%); ^1H NMR (250.1 MHz, $CDCl_3$): $\delta = 0.94$ (t, $^3J_{HH} = 7.3$ Hz, OCH_2CH_3), 1.37 (s, CMe_3), 3.77, 3.78 (2s, $2OCH_3$), 3.99 (q, $^3J_{HH} = 7.3$ Hz, OCH_2CH_3), 4.37, 5.09 (2d, $^3J_{HH} = 10.5$ Hz, 2CH), 5.80 (s, br, NH), 7.34–7.51 (m, H–*Ar*) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$): $\delta = 13.6$ (OCH_2CH_3), 28.4 (CMe_3), 52.2, 52.8 ($2OCH_3$), 53.1, 54.1 (2CH), 61.9 (OCH_2CH_3), 62.8 (CMe_3), 127.5 ($2C_{ortho}$), 128.9 (C_{para}), 129.4 ($2C_{meta}$), 140.9 (C_{ipso}), 161.3, 163.0, 164.3, 168.2, 168.9 (5C=O) ppm.

Minor isomer (11%), ^1H NMR (250.1 MHz, $CDCl_3$): $\delta = 0.93$ (t, $^3J_{HH} = 7.3$ Hz, OCH_2CH_3), 1.34 (s, CMe_3), 3.68, 3.69 (2s, $2OCH_3$), 3.99 (q, $^3J_{HH} = 7.3$ Hz, OCH_2CH_3), 4.27, 5.32 (2d, $^3J_{HH} = 10.5$ Hz, 2CH), 5.71 (s, br, NH), 7.34–7.51 (m, H–*Ar*) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$): $\delta = 13.4$ (OCH_2CH_3), 28.4 (CMe_3), 51.9, 52.7 ($2OCH_3$), 52.9, 53.4 (2CH), 61.9 (OCH_2CH_3), 62.8 (CMe_3), 127.4 ($2C_{ortho}$), 128.8 (C_{para}), 129.5 ($2C_{meta}$), 140.9 (C_{ipso}), 161.4, 162.5, 162.9, 164.1, 168.1 (5C=O) ppm.

Dimethyl 2-[(4-bromo(2-ethoxy-2-oxoacetyl)anilino]-3-[(tert-butylamino)carbonyl]succinate (**5d**, $C_{21}H_{27}BrN_2O_8$)
Brown oil, yield 0.55 g (53%); IR (KBr): $\bar{\nu} = 3375$ (NH), 1742, 1679 (C=O) cm^{-1}.

Major isomer (83%); ^1H NMR (250.1 MHz, $CDCl_3$): $\delta = 1.05$ (t, $^3J_{HH} = 7.0$ Hz, OCH_2CH_3), 1.35 (s, CMe_3), 3.75, 3.76 (2s, $2OCH_3$), 4.03 (q, $^3J_{HH} = 7.0$ Hz, OCH_2CH_3), 4.36, 5.00 (2d, $^3J_{HH} = 10.5$ Hz, 2CH), 5.84 (s, br, NH), 7.24–7.51 (m, H–*Ar*) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$): $\delta = 13.6$ (CH_3), 28.4 (CMe_3), 52.1, 53.0 ($2OCH_3$), 53.2, 54.0 (2CH), 62.2 (OCH_2), 63.0 (CMe_3), 123.0 (C–Br), 129.2, 132.6 (4CH), 140.7 (N–C_{ipso}), 161.0, 162.7, 164.3, 168.0, 168.7 (5C=O) ppm.

Minor isomer (17%); ^1H NMR (250.1 MHz, $CDCl_3$): $\delta = 1.01$ (t, $^3J_{HH} = 7.0$ Hz, OCH_2CH_3), 1.27 (s, CMe_3), 3.69, 3.70 (2s, $2OCH_3$), 4.05 (q, $^3J_{HH} = 7.0$ Hz, OCH_2CH_3), 4.29, 5.21 (2d, $^3J_{HH} = 10.0$ Hz, 2CH), 5.78 (s, br, NH), 7.24–7.51 (m, H–*Ar*) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$): $\delta = 13.6$ (CH_3), 28.4 (CMe_3), 51.8, 52.8 ($2OCH_3$), 53.0, 53.5 (2CH), 61.9 (OCH_2), 62.1 (CMe_3), 122.8 (C–Br), 128.9, 132.5 (4CH), 139.7 (N-C_{ipso}), 161.1, 162.0 , 163.7, 167.8, 168.5 (5C=O) ppm.

Dimethyl 2-[(tert-butylamino)carbonyl]-3-[(2-ethoxy-2-oxoacetyl)-4-nitroanilino]succinate (**5e**, $C_{21}H_{27}N_3O_{10}$)
Yellow powder, mp 110–112°C, yield 0.49 g (51%); IR (KBr): $\bar{\nu} = 3370$ (NH), 1746, 1671 (C=O) cm^{-1}.

Major isomer (81%); ^1H NMR (250.1 MHz, $CDCl_3$): $\delta = 1.11$ (t, $^3J_{HH} = 7.0$ Hz, OCH_2CH_3), 1.41 (s, CMe_3), 3.83, 3.84 (2s, $2OCH_3$), 4.11 (q, $^3J_{HH} = 7.0$ Hz, OCH_2CH_3), 4.44, 5.06 (2d, $^3J_{HH} = 10.5$ Hz, 2CH), 5.78 (s, br, NH), 7.76, 8.31 (2d, $^3J_{HH} = 8.2$ Hz, 4CH-*Ar*) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$): $\delta = 13.6$ (OCH_2CH_3), 28.4 (CMe_3), 52.3, 53.1 ($2OCH_3$), 53.4, 54.0 (2CH), 62.6 (OCH_2CH_3), 63.3 (CMe_3), 124.8, 128.1 (4CH–*Ar*), 147.8, 148.5 (2C–*Ar*), 160.6, 162.2, 164.1, 167.4, 167.6 (5C=O) ppm.

Minor isomer (19%); ^1H NMR (250.1 MHz, $CDCl_3$): $\delta = 1.11$ (t, $^3J_{HH} = 7.0$ Hz, OCH_2CH_3), 1.41 (s, CMe_3), 3.76, 3.77 (2s, $2OCH_3$), 4.11 (q, $^3J_{HH} = 7.0$ Hz, OCH_2CH_3), 4.44, 5.27 (2d, $^3J_{HH} = 10.0$ Hz, 2CH), 5.76 (s, br, NH), 7.63, 8.31 (2d, $^3J_{HH} = 8.2$ Hz, 4CH–*Ar*) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$): $\delta = 13.6$ (OCH_2CH_3), 28.4 (CMe_3), 52.1, 53.1 ($2OCH_3$), 53.5, 54.0 (2CH), 62.6 (OCH_2CH_3), 63.3 (CMe_3), 124.9, 128.0 (4CH–*Ar*), 147.7, 148.6 (2C–*Ar*), 160.5, 162.1, 164.1, 167.5, 167.8 (5C=O) ppm.

Diethyl 2-[(tert-butylamino)carbonyl]-3-[(2-ethoxy-2-oxoacetyl)-4-nitroanilino]succinate (**5f**, $C_{23}H_{31}N_3O_{10}$)
Yellow powder, mp 216–218°C, yield 0.53 g (52%); IR (KBr): $\bar{\nu} = 3385$ (NH), 1745, 1685 (C=O) cm^{-1}.

Major isomer (91%); ^1H NMR (250.1 MHz, $CDCl_3$): $\delta = 1.07$ (t, $^3J_{HH} = 7.3$ Hz, OCH_2CH_3), 1.22–1.34 (m, $2CO_2$-CH_2CH_3), 1.38 (s, CMe_3), 4.07–4.34 (m, $3OCH_2CH_3$), 4.41, 5.01 (2d, $^3J_{HH} = 10.5$ Hz, 2CH), 5.87 (s, br, NH), 7.73, 8.28 (2d, $^3J_{HH} = 7.0$ Hz, 4CH–*Ar*) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$): $\delta = 13.5$, 13.9, 14.0 ($3OCH_2CH_3$), 28.3 (CMe_3), 52.2, 54.1 (2CH), 61.9, 62.4, 62.5 ($3OCH_2CH_3$), 63.2 (CMe_3), 124.7, 128.1 (4CH–*Ar*), 147.0, 147.3 (2C–*Ar*), 160.6, 162.1, 164.5, 167.3, 167.8 (5C=O) ppm.

Minor isomer (9%); ^1H NMR (250.1 MHz, $CDCl_3$): $\delta = 1.07$ (t, $^3J_{HH} = 7.3$ Hz, OCH_2CH_3), 1.22–1.34 (m, $2CO_2$–CH_2CH_3), 1.38 (s, CMe_3), 4.41, 5.20 (2d, $^3J_{HH} = 10.5$ Hz, 2CH), 5.81 (s, br, NH), 7.64, 7.89 (2d, $^3J_{HH} = 7.0$ Hz, 4CH–*Ar*) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$): $\delta = 13.5$, 13.9, 14.0 ($3OCH_2CH_3$), 28.3 (CMe_3), 51.9, 53.7 (2CH), 62.0, 62.3, 62.5 ($3OCH_2CH_3$), 63.2 (CMe_3), 125.0, 127.7 (4CH–*Ar*), 147.0, 147.3 (2C–*Ar*), 160.6, 161.8, 163.8, 167.3, 167.9 (5C=O) ppm.

Acknowledgements

We are grateful to Dr. *Farzad Nikpour* for his assistance.

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